#### **ASETSDefense 09**



#### Cadmium Coating Alternatives for High-Strength Steel JTP – Phase II



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### **Project Overview**



### **Objective**

 Assess DoD-selected Cadmium alternatives in accordance with the DoD-approved Joint Test Protocol (JTP) for <u>both traditional plating and brush plating</u> of HSS applications.

(JTP is available at <a href="https://www.jgpp.com">www.jgpp.com</a>)

#### **Approach**

- Two-phased approach:
  - Phase I (HE and adhesion testing): NAVAIR (complete)
  - Phase II (JTP test matrix): AFRL/CTC (complete)
- Down-selection of candidates after each phase of testing is complete (Phases I & II)





### **Project Team Members**



- AFRL Dr. Elizabeth Berman
- CTC Mr. Clayton Drees, Ms. Leanne Debias
- NAVAIR Mr. Steve Brown
- Boeing Mr. Joe Osborne
- ARL Mr. Brian Placzankis
- WMTR Mr. Jay Curry
- Hill AFB Mr. Nate Hughes
- Alumiplate Mr. Gus Vallejo
- Marshall Labs Mr. John Marshall





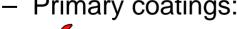
### **Phase I Overview and Selection Process**

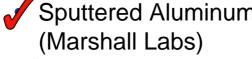


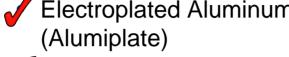


- Primary coatings:
  - Sputtered Aluminum (Marshall Labs)
  - **Electroplated Aluminum** (Alumiplate)

    - Sn-Zn (Dipsol)
- Tests Conducted in Phase I
  - -Hydrogen Embrittlement
  - -Re-embrittlement
  - -Adhesion
- **Selection Process** 
  - WebEx Teleconference to review results
  - -E-mail voting to determine Phase II candidates
  - Down-selected coating for Phase II









Acidic Zn-Ni (Boeing, Seattle)

Repair coatings:

Brush Zn-Ni (SIFCO 4018)

Brush Sn-Zn (LDC 5030)

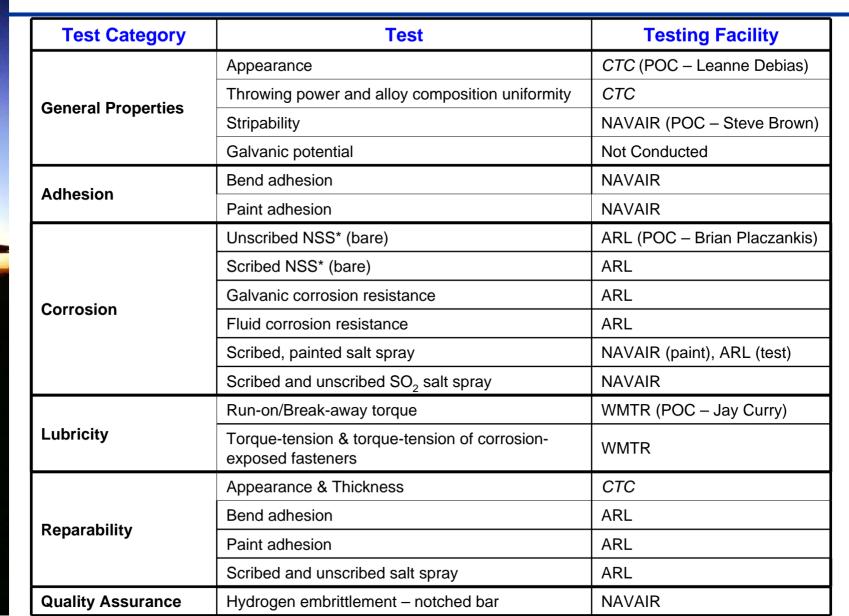
Spray Aluminum-ceramic (Sermetel 249/273)





#### **Phase II Tests**









## **Primary Coating Appearance Test Results**



Coating	Appearance Results
LHE Cadmium (Baseline) – Hill AFB	Coating is continuous but not uniform, showing some edge effect; coating is smooth, adherent, and free from blisters, pits, excessive powder, and contamination
IVD Aluminum (Baseline) – Hill AFB	Coating is continuous, uniform, smooth, adherent, and free from blisters, pits, excessive powder, and contamination
IVD Aluminum (Baseline) – Cametoid Technologies	Coating is continuous, uniform, smooth, adherent, and free from blisters, pits, excessive powder, and contamination
LHE Zinc-Nickel (Dipsol IZ-C17)	Coating is continuous but not uniform also containing a few spots of possible contamination; otherwise, the coating is smooth, adherent, and free from pits, blisters, and excessive powder
Electroplated Aluminum	Coating is continuous, uniform, smooth, adherent, and free from blisters, pits, excessive powder, and contamination
Sputtered Aluminum	Coating is continuous, uniform, smooth, adherent, and free from blisters, pits, excessive powder, and contamination

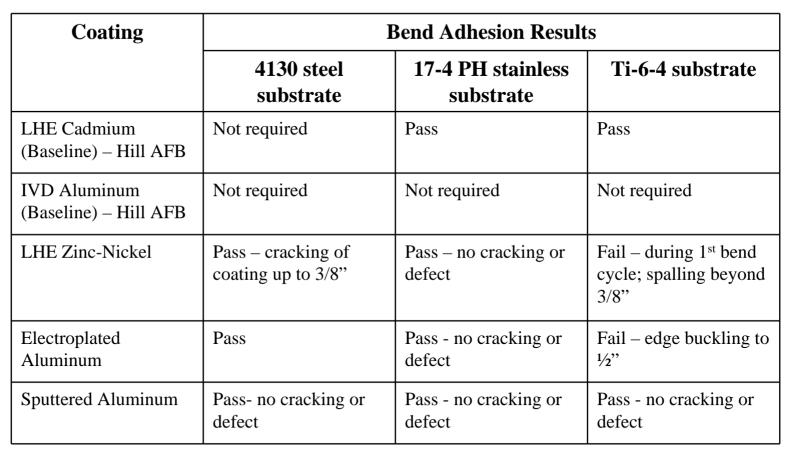
All Primary Coatings Passed Appearance Testing





## Primary Coating Bend Adhesion Test Results





#### Mixed Bend Adhesion Results

- •All primary coatings passed using both 4130 steel & 17-4 stainless
- •LHE Zinc-Ni and electroplated Al failed bend adhesion for Ti-6-4 due to spalling and edge buckling respectively





## **Primary Coating Bend Adhesion Test Results**







Zinc-Nickel on Ti-6-4



Electroplated Aluminum on Ti-6-4



## **Primary Coating Paint Adhesion Results**



	Paint Adhesion						
Coating	(1, 4, 7 day duration)						
	MIL-PRF-23377, Class C2	MIL-PRF-85582, Class C1	MIL-PRF-85582, Class N				
LHE Cadmium (Baseline) – Hill AFB	Pass	Pass	Pass				
	all durations	all durations	all durations				
IVD Aluminum (Baseline) – Hill AFB	Pass	Pass	Pass				
	all durations	all durations	all durations				
IVD Aluminum (Baseline) – Commercial Vendor	N/A	N/A	N/A				
LHE Zinc-Nickel	Pass	Fail	Pass				
	all durations	4 & 7 day duration	all durations				
Electroplated Aluminum	Pass	Pass	Pass				
	all durations	all durations	all durations				
Sputtered Aluminum	Pass	Pass	Pass				
	all durations	all durations	all durations				

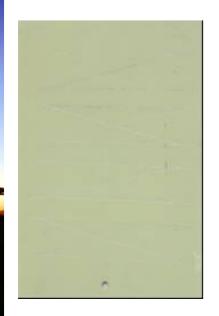
• Primary coatings passed paint adhesion testing with one exception (LHE Zinc-Ni failed for 85582 Class C1 after 4 and 7 days)



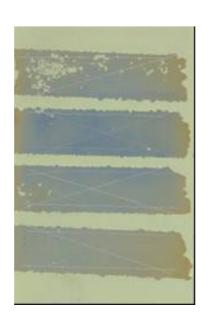


## **Primary Coating Paint Adhesion Results**

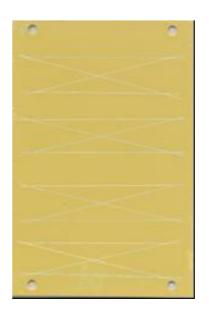




IVD Al with MIL-PRF-85582 C1, after 4 days



LHE Zn-Ni with MIL-PRF-85582 C1, after 4 days



Electroplated Al with MIL-PRF-23377, after 7 days



Sputtered Al with MIL-PRF-23377, after 4 days



### **Primary Coating** Neutral Salt Spray Corrosion Results U.S. AIR FORCE



Coating	Neutral Salt Spray C	orrosion Resistance	
	Unscribed Panels (maximum exposure 3000 hours)	Scribed Panels (maximum exposure 3000 hours)	
LHE Cadmium (Baseline) – Hill	Pass	Pass	
AFB	no damage – rating of 9 at termination	no damage – rating of 9 at termination	
IVD Aluminum (Baseline) – Hill	Fail	Fail	
AFB	excessive rusting – rating of 0 at termination	excessive rusting – rating of 0 at termination	
LHE Zinc-Nickel	Pass	Pass	
	sacrificial coating breakdown only - rating of 9 at termination	sacrificial coating breakdown only - rating of 9 at termination	
Electroplated Aluminum	Fail - marginal	Fail	
	chromate depletion and pin hole formation – rating of 7 at termination	sacrificial coating breakdown and rust – rating of 0 at termination	
Sputtered Aluminum	Fail	Fail	
	excessive rusting – rating of 0 at termination	excessive rusting – rating of 0 at termination	

Only LHE Zinc-Nickel performed as well as the Cd baseline in neutral salt spray corrosion resistance





## Primary Coating Galvanic Corrosion Resistance



<b>Galvanic Corrosion Resistance</b>
<b>Average Change in Conductivity (milliohms)</b>

Average change in conductivity (millioning)									
	Disale	4130 V	Vasher	17-4PH Washer		CuBe Washer		AlNiBr Washer	
Coating	Block Substrate	Salt Spray	Cyclic	Salt Spray	Cyclic	Salt Spray	Cyclic	Salt Spray	Cyclic
No coating	2024 AI	88.0	N/A	52.7	2.6	31.3	0	0	0
	7075 AI	68.3	N/A	270.0	N/A	12.6	0	3.9	0
LHE Cadmium (Baseline)	2024 AI	0	0	0.43	0.37	0	0	0	0.47
(Daseille)	7075 AI	0	0	0.27	0.47	0	0	0	0
IVD Aluminum	2024 AI	0	0	0.03	0	0	0	0	0
(Baseline)	7075 AI	0	0	0	0	0	0	0	0
LHE Zinc-Nickel	2024 AI	0.33	0	0.47	0	2.1	0	0.2	0
	7075 AI	0.47	0.03	0.60	0	3.2	0	0.73	0
Electroplated	2024 AI	0.17	0	0	0	0	0	0.47	0
Aluminum	7075 AI	1.53	0	0.60	0	0	0	0.73	0
Sputtered Aluminum	2024 AI	0.1	0	0.07	0	0.07	0	0.07	0
Alullillulli	7075 AI	0	0	0	0	0.17	0.03	0.33	0

• Sputtered Al performed best, followed by electroplated Al and LHE Zn-Ni

Red = Worse than Cd
Blue = Equal to or better than Cd
Green = Marginally worse than Cd





### **Primary Coating** Fluid Corrosion Resistance Results us an



- Cadmium baseline, IVD Al baseline, and LHE Zinc-Nickel performed similarly, with measurable weight changes and appearance changes for exposure to paint removers
- Electroplated Al performed better than cadmium and IVD Al for all fluids tested
- Sputtered Al performed worst, with weight changes noted for one paint remover, aircraft deicer, reagent water, and water-saturated lubricant





## Primary Coating Scribed, Painted Salt Spray Results us. A



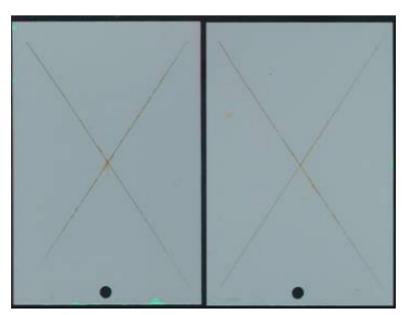
- Three primers tested for a maximum exposure of 3000 hours
- Cadmium performed the best with all 9 ratings after 3000 hours
- LHE Zn-Ni performed similarly to cadmium, with three panels achieving 9 ratings, though two others achieved a 4 and 5 for creepage and pinhole rust
- Electroplated Al lasted 3000 hours but received ratings ranging from 0 – 5
- Sputtered Al and IVD Al both failed to achieve 3000 hours of exposure due to excessive rust





# Representative Images of Scribed, Painted Salt Spray Panels





Cd-plated panels (3000 hours)



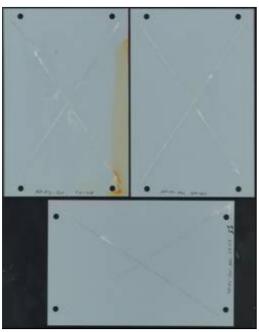
IVD Al Coated Panels (2000 hours)



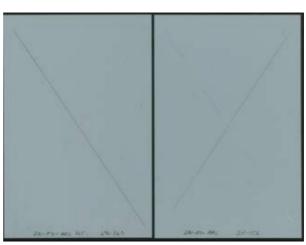


# Representative Images of Scribed, Painted Salt Spray Panels

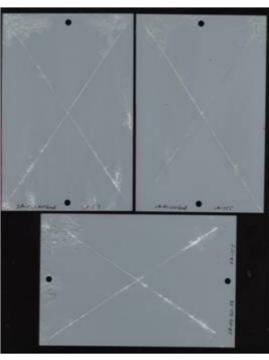




Panels (3000 hours)



LHE Zn-Ni Panels (2000 hours)



Sputtered Al Panels (1500 hours)



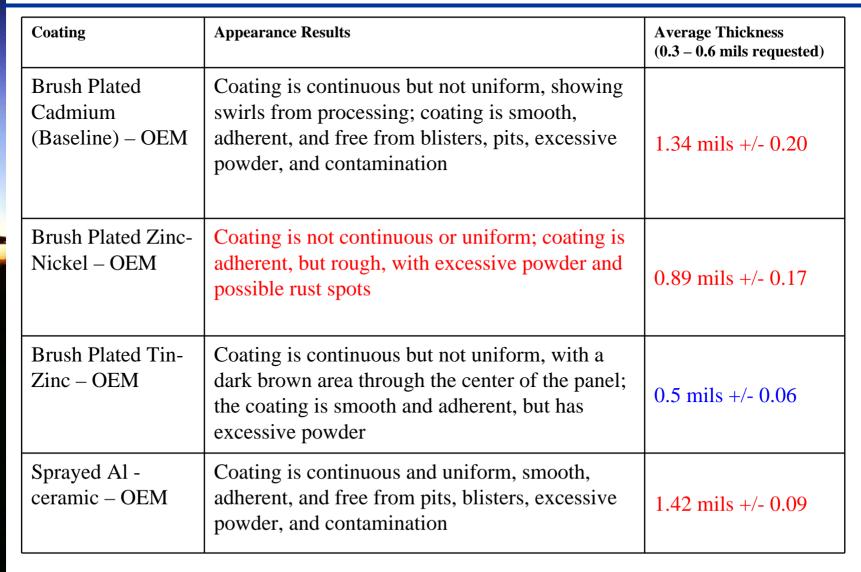


- Reporting Maximum Locking Torque and Breakaway Torque for the 3/8-inch and 5/8-inch fasteners
- 3/8-inch LHE Zinc-Nickel, electroplated aluminum and sputtered aluminum fasteners pass JTP criteria (9.5 inlbs)
- 5/8-inch Cadmium baseline and all coatings fail JTP criteria (32 in-lbs)



## Repair Coating Appearance and Thickness Results









## Repair Coating Bend Adhesion Test Results





Sprayed Aluminum - ceramic

- **Brush Plated Cd (baseline)**
- Brush plated Cd and sprayed Aluminum-ceramic exhibit significant adhesion losses during bend testing
- Brush plated Sn-Zn and Zn-Ni passed adhesion testing
- Results consistent with those observed during Phase I testing





## **Repair Coating** Neutral Salt Spray Corrosion Results U.S. AIR FORCE



Coating	Neutral Salt Spray Corrosion Resistance			
	Unscribed Panels (maximum exposure 3000 hours)	Scribed Panels (maximum exposure 3000 hours)		
Brush LHE Cadmium (Baseline) – Boeing, St. Louis	Pass  no damage – rating of 10 at termination	Pass  no damage – rating of 9 at termination		
Brush Tin-Zinc – Boeing, St. Louis	Fail excessive rusting – termination at 1000 hours	Fail severe rusting – termination at 500 hours		
Brush Zinc-Nickel – Boeing, St. Louis	Pass sacrificial coating breakdown only - rating of 9 at termination	Pass sacrificial coating breakdown – average rating of 7.7 at termination		
Sermetel 249/273 – Boeing, St. Louis	Fail excessive rusting – termination at 500 hours	Fail excessive rust – termination at 500 hours		

Only Brush Zinc-Nickel performed as well as the Cd baseline in neutral salt spray corrosion resistance





### **Summary**



- Phase II testing has been completed (CTC, NAVAIR, ARL, WMTR)
- <u>Mixed results</u> obtained for primary and repair coatings:
  - Primary Coatings
    - <u>Electroplated Al</u> performed similar to or better than Cd in most tests
      - But failed all neutral salt spray corrosion tests and painted cyclic SO<sub>2</sub> salt spray tests, and exhibited inconsistencies in composition for throwing power tests
    - <u>LHE Zn-Ni</u> performed best in all neutral salt spray tests and in painted cyclic SO2 salt spray tests,
      - But exhibited failures in bend adhesion, paint adhesion, galvanic corrosion resistance, and hydrogen re-embrittlement
    - Overall, <u>sputtered Al</u> performed worst, failing the most tests,
      - But yielded the best galvanic corrosion resistance and bend adhesion results

#### Repair coatings

- Brush plated Zn-Ni performed best overall, but failed appearance tests due to excessive surface roughness
- Brush plated Sn-Zn failed corrosion tests
- Sermetel coating failed adhesion and corrosion tests





## **Back-Up Slides**







- Appearance visual exam
- Throwing power
  - Test fixture surrounds panel, with one access slot
  - Fixture + panel is placed in solution at 3 different orientations
  - Uniformity of coating is measured at 3 locations on each panel
- Strippability
  - Specimens are stripped by vendor-recommended method
  - Half of specimens are tested
  - Remaining specimens are recoated and tested
    - Hydrogen Embrittlement
    - Adhesion
- Galvanic Potential
  - Three types of measurements are performed over 5 days: open circuit potential measurement, electrochemical impedance spectroscopy, and tafel analysis







### Adhesion (primary coatings)

- Bend adhesion
  - Specimen is bent back and forth through 180° until the coating and/or substrate ruptures
- Wet tape paint adhesion
  - Primers are applied to test panels (14 day cure)
    - MIL-PRF-85582 Type I, Class C1
    - MIL-PRF-85582, Type I, Class N
    - MIL-PRF-23377 Type 1, Class C
  - Panels are immersed in distilled water at following conditions:
    - 23°C for 24 hours
    - 49°C for 96 hours
    - 65°C for 168 hours
  - Perform tape adhesion according to ASTM D3359, Method B







### Corrosion (primary coatings)

- Unscribed and Scribed Neutral Salt Spray (bare)
  - Bare panels exposed to a 5% NaCl solution sprayed at 35°C, until coating failure
- Galvanic corrosion resistance
  - Components of test assemblies: 2024 or 7075 Al test block, coated with MIL-PRF-85582, Class 1, Type N, test washer (4 alloys), nuts, bolts, anodized washers
  - Test assemblies are exposed to salt fog for 168 hours and cyclic corrosion for 336 hours
- Fluid corrosion resistance
  - Immerse panels in specified fluid at 100°F for 7 days
  - Test fluids: sea water, deicers, paint removers, cleaners, lubricants (14 total)







### Corrosion (continued)

- Scribed Painted Neutral Salt Spray
  - Test panels are primed with
    - MIL-PRF-85582 Type I, Class C1
    - MIL-PRF-85582, Type I, Class N
    - MIL-PRF-23377 Type 1, Class C
  - Test panels are exposed to 5% NaCl solution at 35°C for 3000 hours or until red rust
- Scribed and Unscribed SO<sub>2</sub> Salt Spray
  - Unpainted panels and scribed, painted panels (same primers as above)
  - Expose to 5% NaCl and SO<sub>2</sub> gas IAW ASTM G85 A4 until coating failure (red rust)







#### Lubricity (primary coatings)

- Run-on/Breakaway Torque
  - Record maximum locking torque after 2 complete turns from point where the top of the nut is flush with the end of the bolt
  - Breakaway torque is measured during removal of the nut
  - Measure for 15 lock/breakaway cycles and examine at 10x for thread damage
- Torque Tension
  - Measure torque and induced load with test fixture for the range of 30%-60% of the bolt UTS
  - Repeat for a total of 5 cycles
- Torque Tension of corrosion-exposed fasteners
  - Assemble bolts/nuts/washers onto an Al test block
  - Torque to 60% of UTS for bolt and exposed to cyclic corrosion for 28 days
  - Measure breakaway torque and compare to unexposed set









- Initial qualification coating applied to bare substrate and tested
- Final qualification candidate primary coating of choice is abraded to generate a bare area and then repaired with a brush plating
- Testing Methods
  - Appearance visual exam
  - Bend adhesion bend specimen back over itself until rupture
  - Thickness cross-section and microscopy
  - Scribed and unscribed salt spray (bare) until failure
  - Paint adhesion apply primers, immerse in distilled water at same temps/times as primary coatings, and perform cross-hatch adhesion according to ASTM D3359, method B
- Quality assurance HE testing to compare to Phase I





#### **Alternative Selection – Phase I**



#### **Down-selected Coatings for Phase II:**

- Primary test coatings
  - LHE Zinc-Nickel (Dipsol IZ-C17)
  - Electroplated Aluminum
  - Sputtered Aluminum
  - Controls:
    - Cadmium
    - IVD AI: Hill AFB flat panels
    - IVD AI: Cametoid fasteners, washers and HE bars
- Repair test coatings (Cd brush control)
  - Aluminum-Ceramic Repair Coating (Sermetel)
  - Zinc-Nickel brush repair
  - Tin-Zinc brush repair
  - Control Cd brush

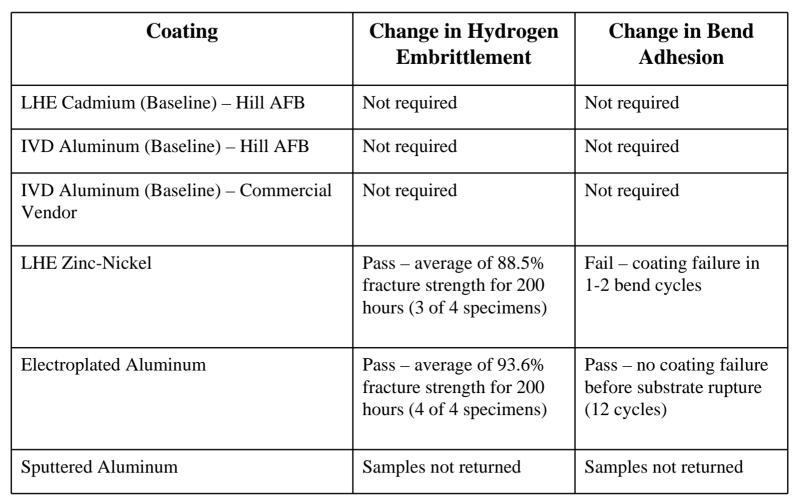
All Phase II testing methods were performed according to the procedures and requirements in the JTP.





## **Primary Coating Stripability Test Results**





#### Mixed stripability results:

- •Electroplated Al passed
- •LHE Zinc-Ni failed due to change in bend adhesion in 1-2 bend cycles





### **Throwing Power Fixture**









- Test coupon is inserted in fixture and entire assembly is submerged in plating solution
- Fixture alignment can be varied to simulate multiple complex geometries
- Composition readings taken every 0.5" down the length of the coupon





## LHE Zinc-Nickel Throwing Power Composition Results



Reading #	Wt %	Wt%	Wt%	Wt%
	Oxygen	Iron	Nickel	Zinc
1	1.6%	0.3%	15.9%	82.2%
2	1.5%	0.3%	16.1%	82.1%
3	1.9%	0.5%	15.7%	81.9%
4	1.9%	1.5%	15.6%	81.0%
5	2.1%	1.7%	15.3%	80.8%
6	1.9%	2.0%	14.1%	82.0%
7	2.0%	2.8%	14.3%	80.8%
8	2.0%	2.8%	14.0%	81.2%
9	1.5%	3.2%	13.6%	81.7%

**Panel #1**: notch in fixture facing upward with open end of fixture facing the node

Reading #	Wt %	Wt%	Wt%	Wt%
	Oxygen	Iron	Nickel	Zinc
1	1.7%	5.6%	11.2%	81.6%
2	2.0%	2.6%	11.9%	83.5%
3	2.2%	1.8%	13.3%	82.7%
4	2.4%	1.5%	14.3%	81.9%
5	2.2%	1.2%	15.1%	81.5%
6	2.3%	0.8%	15.4%	81.5%
7	2.1%	0.8%	15.0%	82.1%
8	2.4%	0.8%	15.2%	81.6%
9	2.6%	1.4%	15.5%	80.5%

Panel #3: notch in fixture in vertical position

Reading #	Wt %	Wt%	Wt%	Wt%
	Oxygen	Iron	Nickel	Zinc
1	1.4%	0.2%	15.9%	82.5%
2	1.7%	0.3%	15.9%	82.1%
3	1.8%	0.3%	15.2%	82.7%
4	1.8%	0.8%	15.4%	82.1%
5	1.7%	1.0%	15.8%	81.6%
6	1.8%	1.3%	14.7%	82.1%
7	1.4%	1.5%	14.9%	82.2%
8	1.7%	1.7%	14.5%	82.1%
9	2.2%	2.2%	14.0%	81.6%
	·			<u> </u>

Panel #2: notch in fixture facing upward with open end of fixture facing away from the node

- Nickel and Iron compositions vary along panel length (trend depends on fixture geometry)
- Oxygen and Zinc compositions are more consistent
- Fixture geometry for Panel #3 resulted in most significant composition variation





## **Electroplated Aluminum Throwing Power Composition Results**



Reading #	Wt %	Wt%	Wt%	Wt%	Wt%
	Oxygen	Aluminum	Chromium	Iron	Nickel
1	11.8%	77.6%	5.2%	2.5%	3.0%
2	10.8%	78.7%	4.7%	2.4%	3.5%
3	10.6%	74.9%	4.8%	2.9%	6.8%
4	9.1%	72.5%	4.4%	3.2%	10.8%
5	11.7%	74.9%	5.0%	3.2%	5.2%
6	12.3%	76.8%	5.2%	3.0%	2.9%
7	12.0%	76.6%	5.6%	3.2%	2.6%
8	11.8%	76.6%	5.7%	3.0%	2.9%
9	10.8%	77.4%	4.3%	3.0%	4.5%

<b>Panel #1</b> :	notch	in	fixture	in	vertical	nosition
raner#1.	поил	ш	HALUIC	ш	vertical	DOSIUOH

Reading #	Wt %	Wt%	Wt%	Wt%	Wt%
	Oxygen	Aluminum	Chromium	Iron	Nickel
1	9.9%	80.5%	3.9%	3.0%	2.7%
2	11.0%	79.8%	3.9%	2.9%	2.4%
3	9.4%	79.7%	3.5%	2.7%	4.8%
4	8.2%	76.8%	2.8%	3.0%	9.2%
5	8.5%	74.6%	3.5%	3.8%	9.7%
6	8.3%	78.6%	3.0%	4.4%	5.7%
7	9.4%	77.3%	3.2%	3.2%	6.9%
8	7.9%	70.9%	2.7%	5.8%	12.7%
9	3.6%	54.4%	0.9%	4.1%	37.0%

Panel #3: notch in fixture facing upward when placed in bath

Reading #	Wt %	Wt%	Wt%	Wt%	Wt%
	Oxygen	Aluminum	Chromium	Iron	Nickel
1	13.3%	76.9%	4.7%	2.6%	2.5%
2	11.5%	78.7%	4.4%	2.7%	2.6%
3	10.2%	80.0%	3.7%	2.8%	3.3%
4	9.0%	79.8%	3.8%	3.2%	4.2%
5	8.7%	81.8%	3.1%	3.3%	3.2%
6	11.1%	80.0%	3.8%	3.1%	2.0%
7	9.3%	80.4%	3.9%	3.0%	3.5%
8	10.1%	78.8%	3.9%	2.8%	4.4%
9	10.8%	77.2%	2.9%	2.8%	6.3%

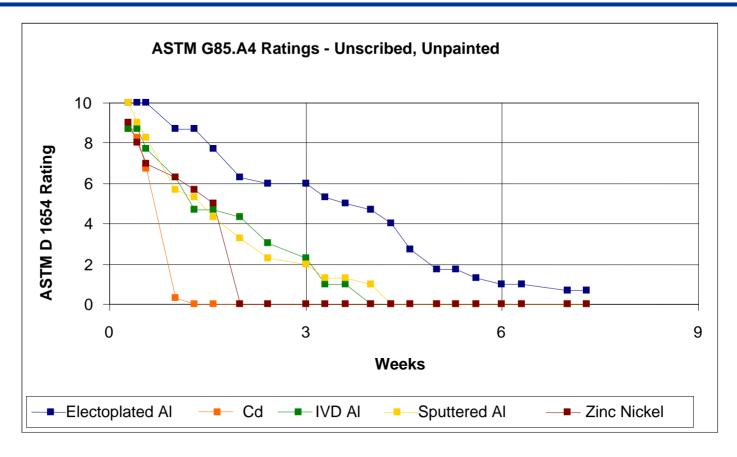
Panel #2: notch in fixture facing downward when placed in bath

- Oxygen, Aluminum, and Nickel compositions varied considerably for each panel
- No apparent trends in data
- Final readings for Panel #3 represent significant deviations in coating composition



# Primary Coating SO<sub>2</sub> Salt Fog Corrosion Resistance Unscribed, Unpainted Specimens





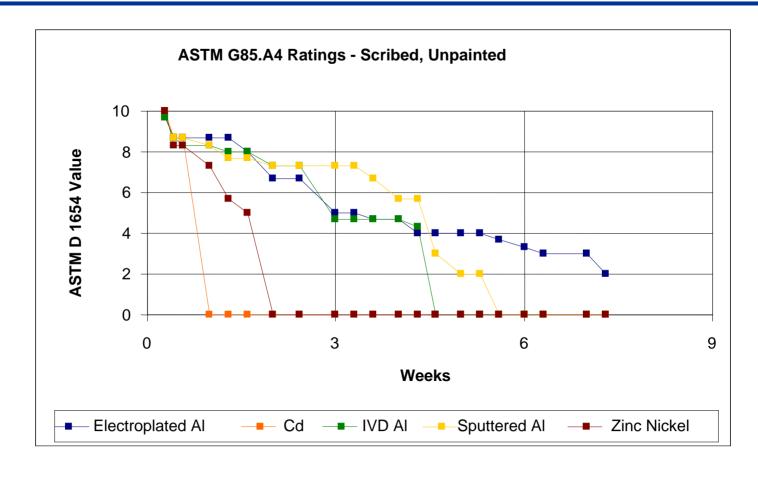
- All primary coatings outperformed baseline Cd
- Aluminum coatings yielded the best results, with electroplated aluminum showing the highest level of corrosion resistance





# Primary Coating SO<sub>2</sub> Salt Fog Corrosion Resistance Scribed, Unpainted Specimens





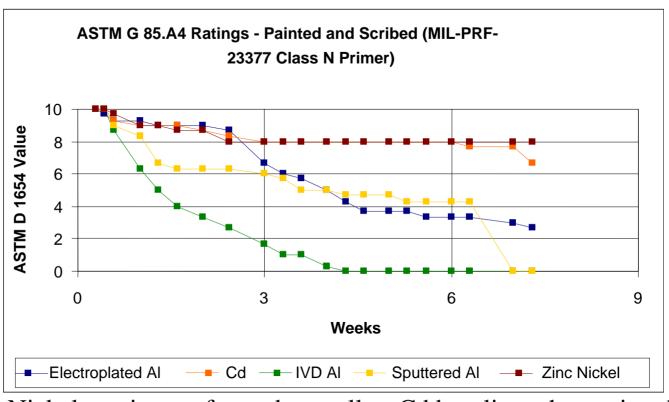
- All primary coatings outperformed Cd in scribed testing
- Zinc-Nickel coating was significantly less effective than the aluminum coatings





# Primary Coating SO<sub>2</sub> Salt Fog Corrosion Resistance Scribed, Painted Specimens





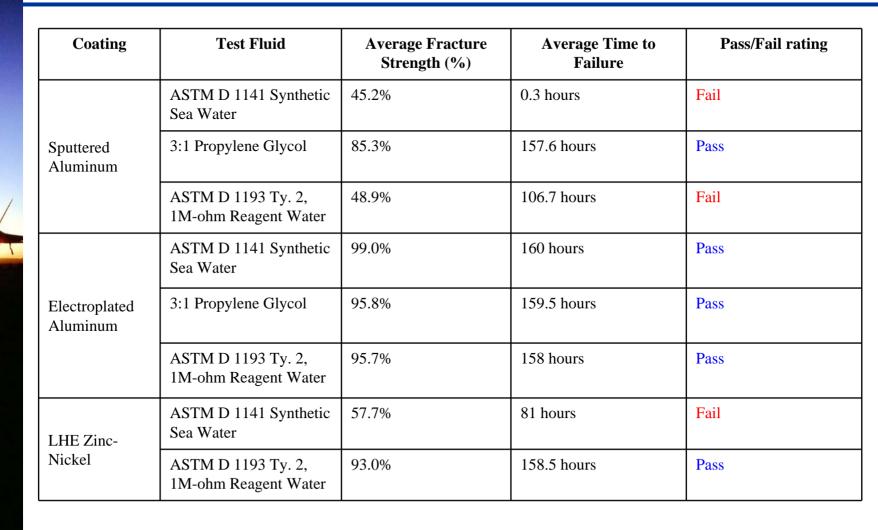
- Zinc-Nickel coating performed as well as Cd baseline when painted with MIL-PRF-23377 Class N non-chromated primer
- Aluminum based coatings were less effective, with IVD Al baseline displaying the poorest results
- Results were similar for MIL-PRF-23377 Class C2 chromated primer and MIL-PRF-85582 Class N non-chromated primer





## Primary Coating Hydrogen Re-Embrittlement Test Results





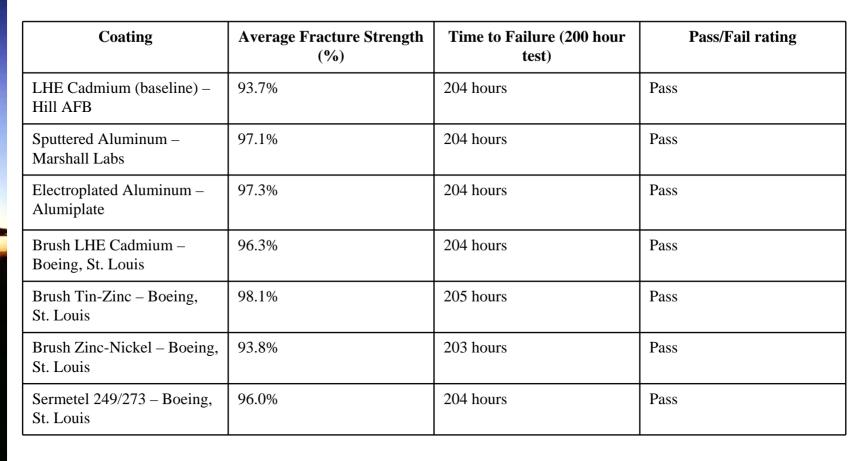
• Only electroplated aluminum passed all conditions





# Primary / Repair Coating Hydrogen Embrittlement Quality Assurance





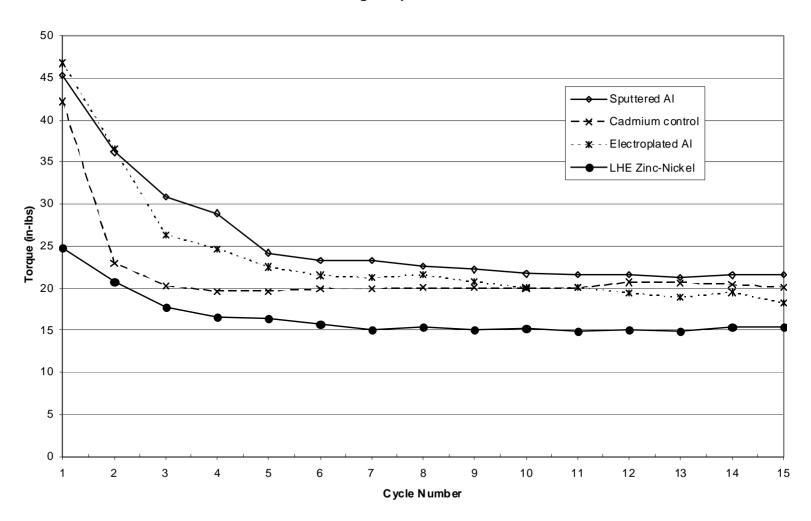
- LHE Zn-Ni tested for hydrogen re-embrittlement only in Phase II
- IVD Al not tested for hydrogen embrittlement in Phase II







#### Maximum Locking Torque for 3/8-inch Fasteners

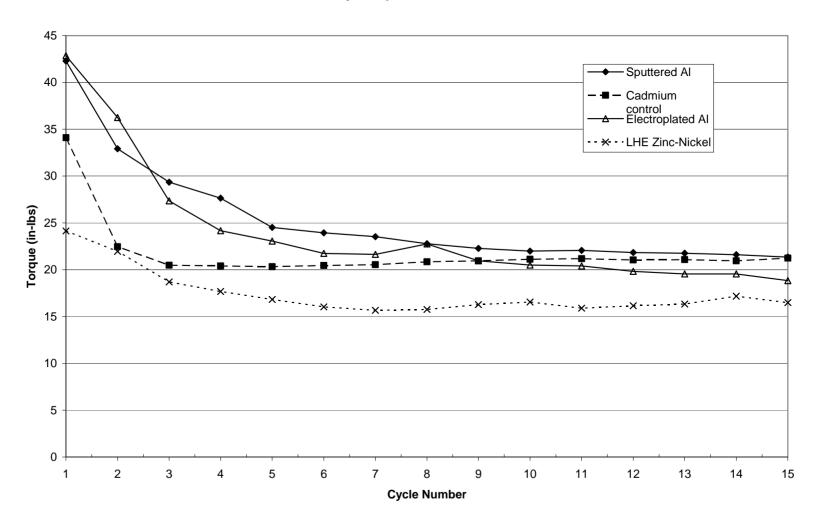








#### **Breakaway Torque - 3/8-inch Fasteners**

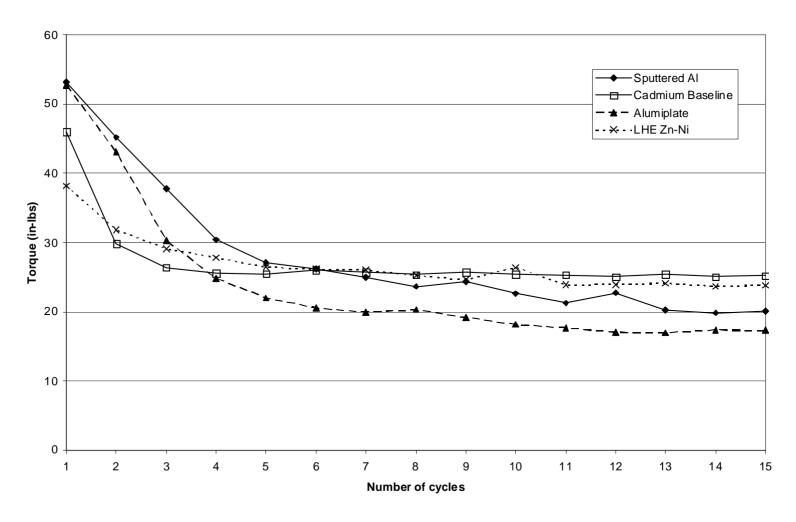








#### **Maximum Locking Torque - 5/8-inch**









#### **Breakaway Torque Results - 5/8-inch fasteners**

